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Internally Sealed Concrete

Monahans, Texas

dP Demonstration Project No. 49
Report No. FHWA-DP-49-2
Interim Report
January 1982

COMPLETED

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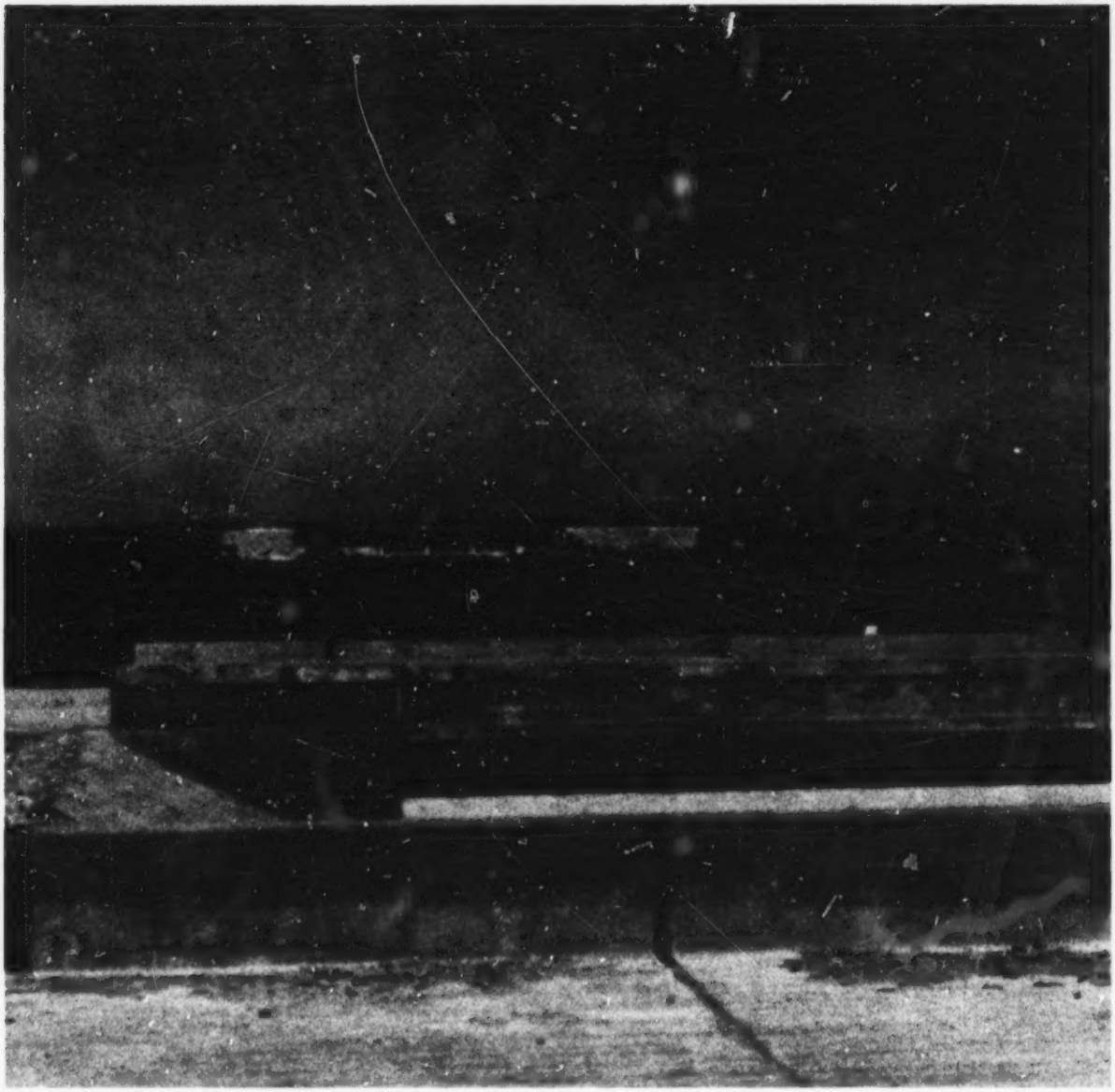
U.S. Department of Transportation
Federal Highway Administration

Eastern Direct Federal Division
Demonstration Projects Program



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16. Abstract

This report describes the construction, heat treatment, and evaluation of an internally sealed concrete bridge deck on IH-20 near Monahans, Texas. The deck contained wax beads throughout its full depth, however, only the top two inches were heated to internally seal the concrete. The overall objective of this study was to obtain additional information on the performance of internally sealed concrete under service conditions.

The heating system and method of heat treatment are described. The tests used to evaluate the effectiveness of the internally sealed concrete are discussed. These include dye penetration, chloride analysis, flexural and compressive, half-cell potential, a crack survey, and a petrographic examination of both the aggregate and the hardened concrete. The results of these tests and observations showed that internally sealing concrete enhances its ability to resist the penetration of chlorides.

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INTERNALLY SEALED CONCRETE

MONAHANS, TEXAS

By

H. D. Butler

Senior Designing Engineer

Texas State Department of Highways & Public Transportation

Tom S. Patty

Geologist

Texas State Department of Highways & Public Transportation

Fred A. Schindler, Jr.

Supervising Concrete Engineer

Texas State Department of Highways & Public Transportation

Demonstration Study No 1-5-79-538

"Internally Sealed Concrete Bridge Decks"

Work Done in Cooperation with
U.S. Department of Transportation
Federal Highway Administration

FHWA Demonstration Project No. 49

"Internally Sealed Concrete"

January 1982

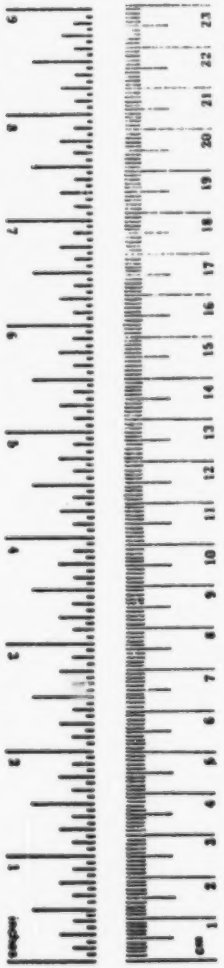
IV

METRIC CONVERSION FACTORS

Approximate Conversions to Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
	LENGTH			
in	inches	*2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
	AREA			
in^2	square inches	6.5	square centimeters	cm^2
ft^2	square feet	0.09	square meters	m^2
yd^2	square yards	0.8	square meters	m^2
mi^2	square miles	2.6	square kilometers	km^2
	acres	0.4	hectares	ha
	MASS (weight)			
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2000 lb)	0.9	tonnes	t
	VOLUME			
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft^3	cubic feet	0.03	cubic meters	m^3
yd^3	cubic yards	0.76	cubic meters	m^3
	TEMPERATURE (exact)			
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius temperature	°C

*1 in = 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price $2.25, SD Catalog No. C13.10:286.



Approximate Conversions from Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
	LENGTH			
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi
	AREA			
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	hectares (10,000 m^2)	2.5	acres	
	MASS (weight)			
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	tonnes (1000 kg)	1.1	short tons	
	VOLUME			
ml	milliliters	0.03	fluid ounces	fl oz
l	liters	2.1	pints	pt
l	liters	1.06	quarts	qt
l	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3
	TEMPERATURE (exact)			
°C	Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F

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INTRODUCTION

Chloride induced corrosion of the top mat of reinforcing steel with the resultant cracking and spalling of surface concrete are considered major causes of the premature deterioration of bridge decks. Through the efforts of the Federal Highway Administration, a method using wax beads was developed to internally seal concrete against the intrusion of deicing salts and other corrosive compounds.

Internally sealed concrete is a standard portland cement concrete modified by replacing a small portion of the aggregate with a like amount of small spherical particles of wax. After the concrete has hardened, the deck is heated to a temperature of approximately 185°F which causes the wax to melt and flow into the pores and capillaries of the concrete. Upon removal of the heat, the wax solidifies and the capillary network becomes a discontinous, closed system that effectively blocks the penetration of water and chlorides.[1]

Objectives

The objectives of this study were to construct a bridge deck impregnated with wax beads, heat treat the deck to internally seal the concrete, and evaluate the performance of the internally sealed concrete deck under service conditions. This study was conducted as a part of the Federal Highway Administration's Demonstration Project 49 "Internally Sealed Concrete".

Description of Test Bridge

The experimental bridge is located approximately five miles east of Monahans, Texas, and carries the west bound lanes of Interstate Highway 20 over Texas Park Road 41. The 1979 Average Daily Traffic was 4250 vehicles per day with 19% trucks.

The bridge is 160'-0" long with three prestressed concrete girder spans (40'-80'-40') and a 7.5 inch thick deck slab. The girders are simply supported and the slab is continuous over the interior bents. The overall width of the bridge is 44.0 feet and the clear roadway width is 42.0 feet. The rail is a concrete parapet type and was not constructed until after the heat treatment was completed.

CONSTRUCTION

Internally sealed concrete has generally been placed as a two to three inch overlay over conventional bridge deck concrete, although it has been placed full depth on some experimental projects. The overlay method is very labor intensive, requiring compaction of the bottom course before applying and finishing the overlay, or cleaning the bottom course surface and coating it with grout if the overlay is not placed immediately. Additionally, the contractors experience and ability to successfully perform an overlay construction must be considered.[1] After consideration of these and other factors, it was decided to use the full depth method for this project.

Materials for Concrete

The materials used in the Class C internally sealed concrete included Type II portland cement from Southwestern Portland Cement Company of Odessa, Texas, fine and coarse aggregates from Trans-Pecos Materials Company, wax beads from Concrete Additives Corporation, Wayne, Pennsylvania, and a set retarding agent.

A petrographic examination was made of the aggregates. It was found that the fine aggregate consisted of Rhyolite fines and siliceous quartz sands. The coarse aggregate was made up of Rhyolite and similar extrusive volcanic rock types.

Test Slab

Prior to placing any bridge deck concrete, a 6'-0" x 8'-0" x 8" thick test slab was cast by the contractor. This test slab was used to

evaluate mix design, construction and vibration procedures, wax bead distribution, and the effects of texturing and curing. The batch design work sheet for both the test slab concrete and the bridge deck concrete is shown in Appendix B. The final batch weights per cubic yard were:

Material	Quantity
Fine Aggregate	935 lbs
Coarse Aggregate	1831 lbs
Cement	611 lbs
Water	32.5 gals
Wax Beads	110 lbs
Retarding Agent	210 ozs

Mixing and Placing Concrete

The concrete was mixed in seven cubic-yard batches at a portable batch plant located approximately five miles from the bridge site. The wax beads were preweighed for each batch and mixed with the fine aggregate. All dry materials were moved up to an elevated hopper by conveyor belt then dumped into a transit-mix truck. Water was added to the dry mix and the concrete mixed with 70 revolutions of the drum at the plant and 25 to 40 revolutions at the bridge. This procedure worked very well. The wax beads did, however, tend to make the conveyor belt slick. This required a little more time to move the sand-bead mix up to the hopper than was required to move the sand alone. The presence of wax beads in the concrete was verified at the job site by mixing a small quantity of fresh concrete and water in a transparent container, vigorously shaking the container, and visually checking for beads on the surface of the water.

There were no problems encountered during mixing and placing the wax bead concrete. Placement of the deck slab began about 11:30 in the morning and was completed at 5:00 in the afternoon. The concrete was placed on the deck with a crane and dumping bucket then finished with a transverse finishing machine. No problems were encountered during finishing and texturing the wax bead concrete and a good riding surface was obtained.

HEAT TREATMENT

Heat treatment of the deck was accomplished using the electric blanket heating system owned by the Federal Highway Administration (FHWA). This system is described in FHWA Implementation Package 77-9 "Internally Sealed Concrete - Guide to Construction and Heat Treatment". Personnel from the FHWA Demonstration Projects Division operated and monitored the heating equipment during the entire heating operation.

Heating System

The heating system consisted of the electric blankets, a 250 kw AC generator, a fused transformer, a control panel, and connecting cables. Other miscellaneous equipment and materials included a portable 110-volt AC generator, electric drill, portable lights, 6"x23"x8' bats of unbacked fiberglass insulation, plastic netting to hold the insulation in place during windy conditions, and plastic sheeting to protect the blankets in the event of rain or snow.

The basic elements of the heating system are shown in Figures 1 and 2. In Figure 1 the 250 kw generator is shown in the background with the transformer in the foreground. The control panel and electric blankets with insulation and netting in place are shown in Figure 2. Also shown in Figure 2 is the partial heat zone at the end of the blankets to provide a temperature transition area between the heated and unheated areas of the deck.

Heat Treating

Complete heating of the deck required eight heat runs, four on

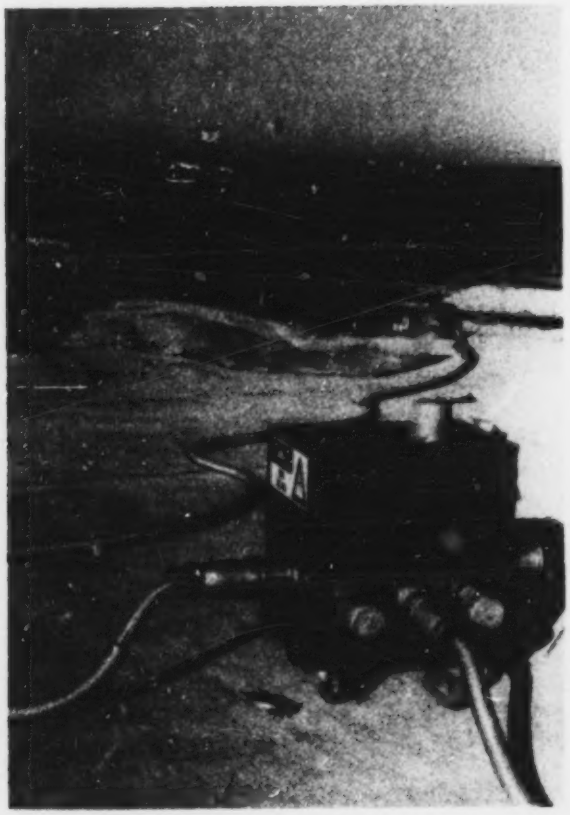

Figure 1. Generator (background) and Transformer

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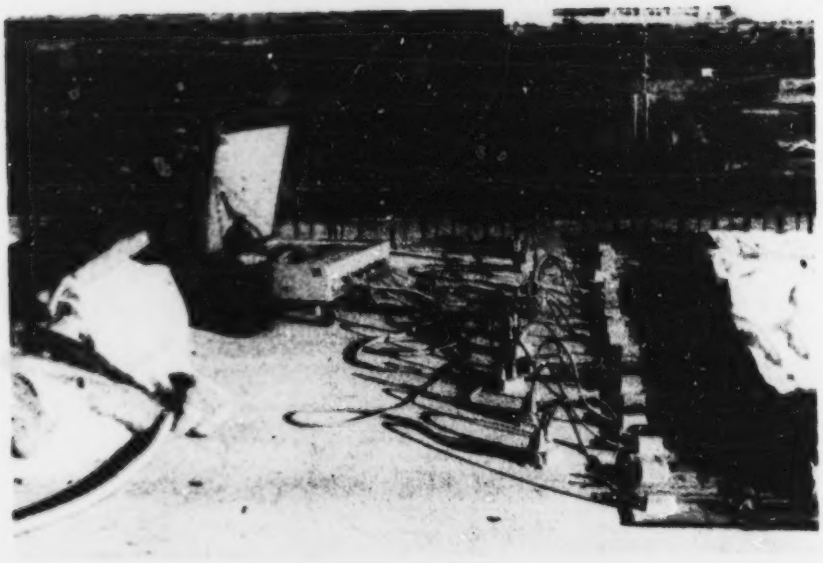

Figure 2. Control Panel, Electric Blankets, and Insulation

Figure 3. Cleaning Deck with Compressed Air

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each side of the bridge. Eighteen blankets were used for each run, 17 full-heat and one partial heat along the inside edge. A partial heat was not needed along the outside edge because the parapet rail was not cast until after the heat treatment was completed. Before beginning a heat run the area to be heated was thoroughly cleaned using compressed air to remove any debris that could cause damage to the blankets (Figure 3).

During heating the concrete temperature was monitored at six locations -- three at the concrete surface and three at a depth of two inches. The surface temperature was monitored by a sensor consisting of a thermocouple embedded in an aluminum disk called a "hockey puck" because of its similar configuration. The temperature at the two-inch depth was monitored by a thermocouple probe encased in a phenolic insulating sleeve. These probes were placed in 1/2-inch diameter holes and packed with drilling dust to provide a good contact with the concrete and to insulate the probes from the high surface temperatures. These six sensors, plus one to monitor ambient air temperature, were connected to a readout panel. Temperatures were recorded each 15 minutes for the first 1 1/2 hours and each 30 minutes thereafter until the desired temperature at the two-inch depth was reached.

Heating began October 27, 1980 at 5:05 pm and was completed October 30, 1980 at 8:30 pm. The average time for the eight runs was 6.4 hours with the shortest being 6 hours and the longest being 6.75 hours. A typical time versus temperature graph is shown in Figure 4.

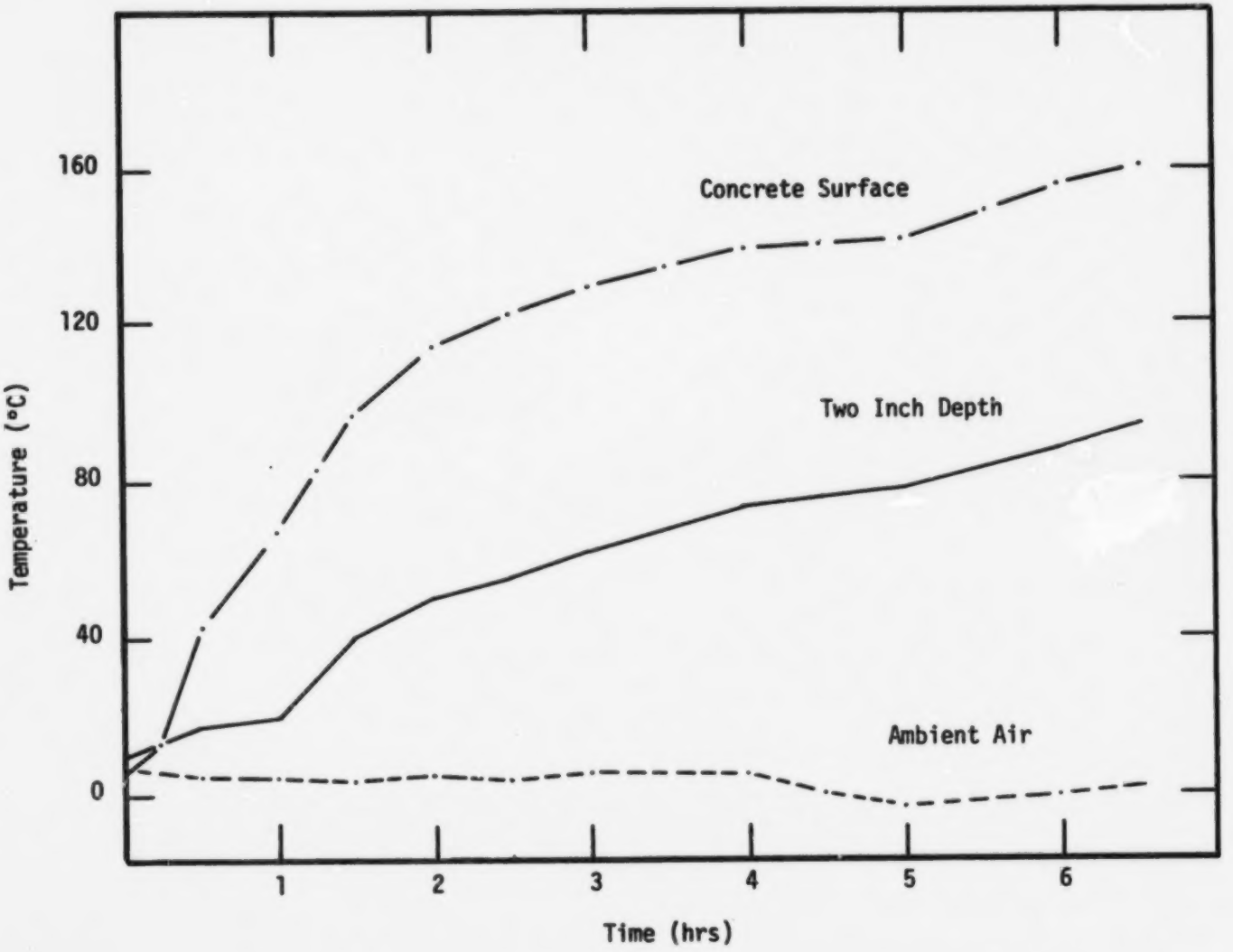

Figure 4. Typical Heat Run Showing Temperature vs. Time Effects

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Approximately one hour was required between runs to move the blankets and prepare for another run. Table 1 shows the times for each run along with the ambient air temperature range and weather conditions.

The deck was heated in a stair-step fashion as shown in Figure 5. Heat was applied and controlled according to the procedure outlined in the FHWA Guidelines for Heat Treating Internally Sealed Concrete and shown in Appendix C. This procedure was designed to permit potentially damaging moisture vapor to escape before the wax starts to block capillaries and cause damaging pressure buildups.

No significant problems were encountered during the heating operation. A strong wind was blowing during the first heat run but the plastic netting did a good job of keeping the insulation in place. Light snow fell during part of the second heat run but the buildup of water never became great enough to require shutting the system down.

Cost Analysis

The contract price for Class "C" internally sealed reinforced concrete bridge slab was $2.60 per square foot more than for the ordinary Class "C" reinforced concrete slab on the project. The following additional costs were incurred when the slab was heat treated:

Generator Rental	$1691.
Fuel for Generator	632.
Insulation	1214.
Plastic Sheeting	48.
Salaries and Wages	3984.
Travel	496.
Miscellaneous Supplies	341.
	$8406.*

*This represents a cost of $1.19 per square foot

The cost of the internally sealed concrete deck was $3.79 per square foot above that for a conventional concrete deck. This does not include the cost of personnel and equipment furnished by the FHWA.

TABLE 1. General data for each heat run

Run No.	Date	Begin Time	End Time	Total Time (hrs)	Air Temp. Range (°C)	Weather Conditions
1	10-27-80	5:05 pm	11:05 pm	6	9-20	Clear, 30 mph wind
2	10-28-80	8:00 am	3:30 pm	6.5	1-5	Windy, overcast, snow
3	10-28-80	4:30 pm	11:15 pm	6.75	-3-9	Overcast, light wind
4	10-29-80	7:45 am	2:30 pm	6.75	2-18	Clear, cold
5	10-29-80	3:15 pm	9:25 pm	6.2	7-15	Clear
6	10-29-80	10:15 pm	4:30 am	6.25	0-6	Clear, calm
7	10-30-80	7:45 am	1:45 pm	6.25	0-19	Clear, calm
8	10-30-80	2:30 pm	8:30 pm	6.0	10-22	Clear, light wind

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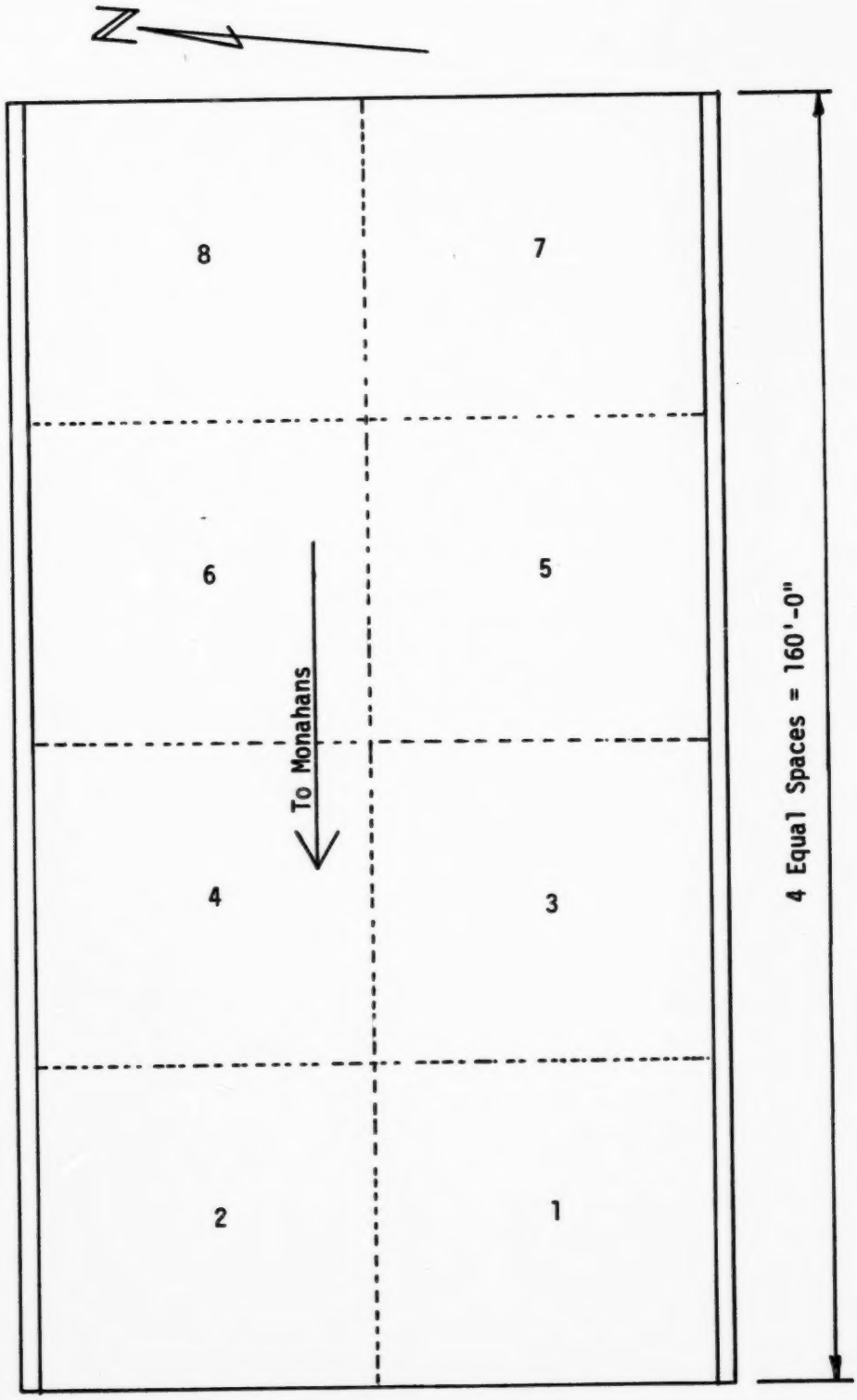

Figure 5. Plan View of Deck Showing Sequence of Heating

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TESTS PERFORMED

Dye Penetration

Dye penetration tests were performed on three two-inch diameter cores taken from the deck approximately two months after heating and on one four-inch core taken from the unheated test slab. These dye penetration tests were performed in accordance with the FHWA "Dye Penetration Test" shown in Appendix D. Without variation, none of the split cores (bridge deck and test slab) that were immersed in a solution of red dye, 1.5% Calgon and water, showed any absorption, even at the four-inch depth where the wax had not been melted.

Freeze-Thaw

The rapid dry freeze-wet thaw procedure of ASTM C 666 was used to test the durability of four-inch diameter cores taken from the heated deck and the unheated test slab. The dynamic modulus of elasticity of each core was measured before freeze-thaw testing began. Subsequent measurements, however, were not possible because the specimens would not respond to being driven to any measurable resonant frequency. Total dynamic modulus loss occurred before 80 cycles were completed.

The freeze-thaw cycling was continued to 300 cycles. The cores did not show any distress in the melted wax zone. One core had severe spalling below the 2-3" depth and cracked horizontally across at this depth. At 255 cycles, three cores had severe spalling around the bottom 3" but did not sustain damage in the upper half of the core through 300 cycles.

The test slab cores had slight surface spalling before 85 freeze-thaw cycles and the cores were breaking up at 120 cycles. Testing was stopped at 144 cycles because cores had been reduced to rubble.

Chloride Penetration

The chloride penetration test was performed on both the bridge deck cores and the test slab cores. One core, No. 4, from each condition was used for the baseline chloride content and was not ponded with sodium chloride. The 90 day ponding procedure and chloride analysis for three 1/2-inch layers, 1/16" to 0.5", 0.5" to 1" and 1" to 1-1/2" was the same as outlined in FHWA's Test "Resistance of Concrete to Chloride Ion Penetration" shown in Appendix D. The results of the chloride analyses are shown in Table 2 for the heat treated bridge deck cores and Table 3 for unheated test slab cores. Figures 6 and 7 show the baseline and total chloride content of these cores graphically.

Crack Survey

The deck was examined before and immediately after heating and no cracks were found that could be related to the heat treatment. There was one transverse crack across the deck over each interior bent but this is typical for deck slabs placed continuously over interior bents. Another survey was made on July 30, 1981, approximately nine months after heating, and the results of this survey are shown in Figure 8. The bridge has not been opened to traffic at this time and these cracks are believed to be the result of shrinkage.

TABLE 2: Chloride Analysis of Concrete Cores (Heat Treated)

Material: Non-Air-Entrained Internally Sealed Concrete (Heat Treated)

Specimen Location: Top 1/2" Level = 1 (1/16" to 0.5" from top surface)

Middle 1/2" Level = 2 (0.5" to 1.0" from top surface)

Bottom 1/2" Level = 3 (1.0" to 1.5" from top surface)

Determinations - Chloride Ion Content:

Specimen Level	1			2			3		
Content	%	PPM	lb/cu yd	%	PPM	lb/cu yd	%	PPM	lb/cu yd
Core #1-Wax									
Total	0.15	1524	6.1	0.03	258	1.0	0.02	160	0.6
Baseline*	0.05	528	2.1	0.02	160	0.6	0.01	86	0.3
Absorbed	0.10	996	4.0	0.01	98	0.4	0.01	74	0.3
Core #2-Wax									
Total	0.15	1524	6.1	0.04	406	1.6	0.02	172	0.7
Baseline*	0.05	528	2.1	0.02	160	0.6	0.01	86	0.3
Absorbed	0.10	996	4.0	0.02	246	1.0	0.01	86	0.4
Core #3-Wax									
Total	0.10	971	3.9	0.02	184	0.7	0.01	135	0.5
Baseline*	0.05	528	2.1	0.02	160	0.6	0.01	86	0.3
Absorbed	0.05	443	1.8	0.00	24	0.1	0.00	49	0.2

*Baseline values determined from Core #4-Wax (Not ponded with chloride).

Note: Weight of chloride per cubic yard based on concrete density of 4000 lb/cu yd.

TABLE 3: Chloride Analysis of Concrete Cores (Unheated)

Material: Test Slab Concrete (Wax Bead Unheated)

Specimen Location: Top 1/2" Level = 1 (1/16" to 0.5" from top surface)

Middle 1/2" Level = 2 (0.5" to 1.0" from top surface)

Bottom 1/2" Level = 3 (1.0" to 1.5" from top surface)

Determinations - Chloride Ion Content

Specimen Level	1			2			3		
Content	%	PPM	lb/cu yd	%	PPM	lb/cu yd	%	PPM	lb/cu yd
Core #1-TSWB Total	0.26	2579	10.3	0.07	676	2.7	0.03	332	1.3
Baseline*	0.04	356	1.4	0.02	184	0.7	0.02	160	0.6
Absorbed	0.22	2223	8.9	0.05	492	2.0	0.01	172	0.7
Core #2-TSWB Total	0.27	2696	10.7	0.06	586	2.3	0.03	283	1.1
Baseline*	0.04	356	1.4	0.02	184	0.7	0.02	160	0.6
Absorbed	0.23	2340	9.3	0.04	402	1.6	0.01	123	0.5
Core #3-TSWB Total	0.25	2462	9.8	0.07	700	2.8	0.02	234	0.9
Baseline*	0.04	356	1.4	0.02	184	0.7	0.02	160	0.6
Absorbed	0.21	2106	8.4	0.05	516	2.1	0.00	74	0.3

*Baseline values determined from Core #4-TSWB (Not ponded with chloride).

Note: Weight of chloride per cubic yard based on concrete density of 4000 lb/cu yd.

NON-AIR ENTRAINED INTERNALLY SEALED CONCRETE (HEAT-TREATED)

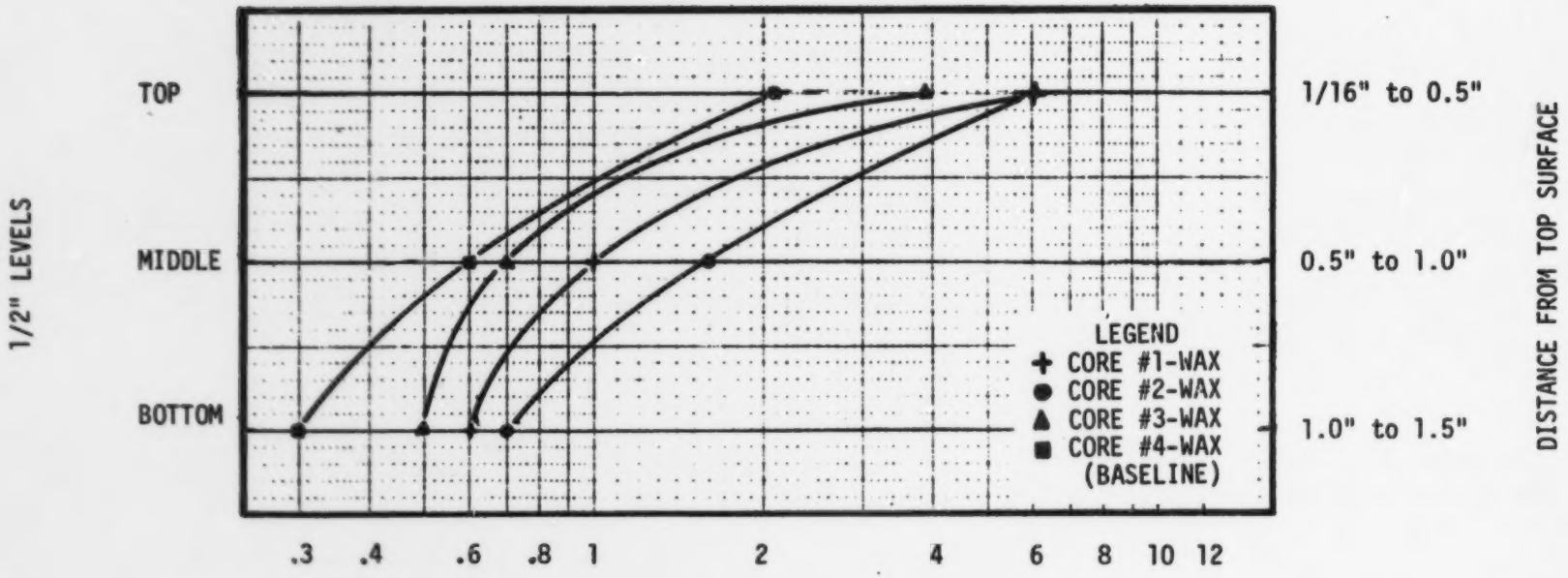

Figure 6

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TEST SLAB CONCRETE (WAX BEAD UNHEATED)

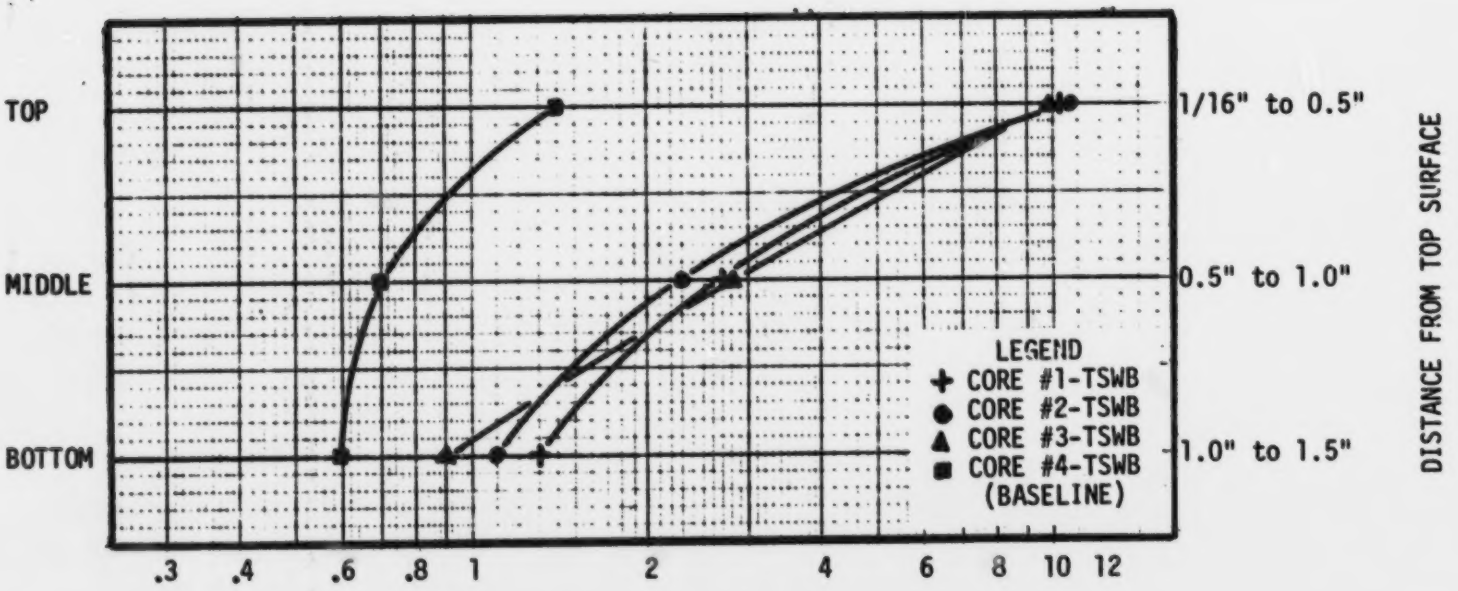

Figure 7

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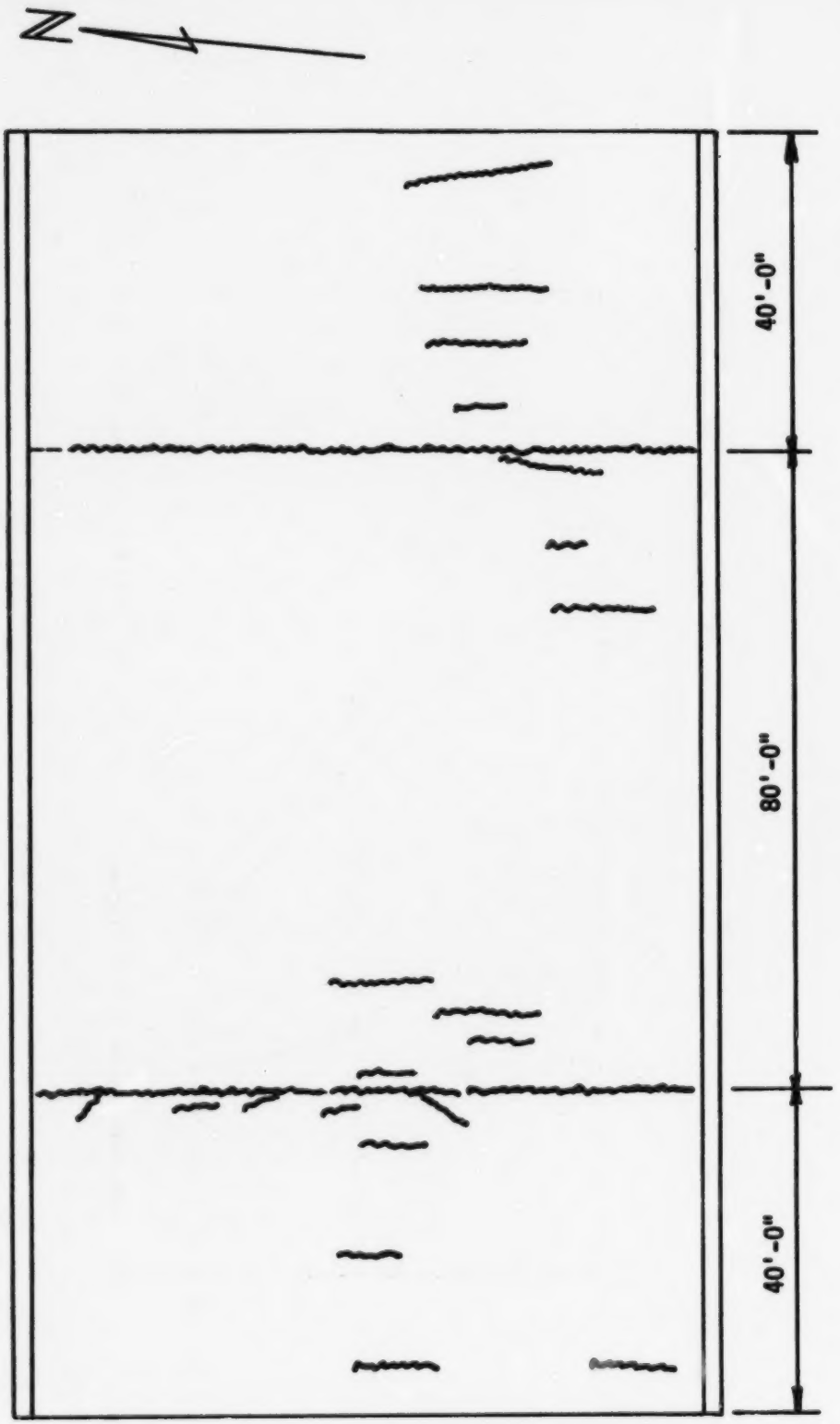

Figure 8. Plan View of Deck Showing Results of Crack Survey Made July 30, 1981

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Strength

The flexural strength of the concrete for both the test slab and the bridge deck was determined from bending tests performed on 15 6"x6"x20" beams, three from the test slab and 12 from the bridge deck. The concrete was seven days old at the time of testing. The range of strengths for the test slab was from 550 psi to 665 psi with an average of 612 psi. The beams from the deck concrete ranged from 600 psi to 738 psi with an average of 684 psi. With the exception of one beam from the test slab that broke at 550 psi, all other beams tested greater than the minimum required seven-day strength of 550 psi.

In addition to the flexural strength tests, four cores from the test slab were tested in compression at 37 days and one was tested at 97 days. At 37 days the compressive strength ranged from 3840 psi to 4469 psi with an average of 4125 psi. The core tested at 97 days had a compressive strength of 4440 psi. Nine cylinders were made at the time of deck placement and tested approximately 13 months later. The compressive stress ranged from 4754 psi to 5124 psi with the average being 4959 psi.

Half-Cell

Approximately nine months after the bridge deck was heated, a half-cell survey was conducted. The bridge was not open to traffic and no deicing salts had been applied. All measured half-cell potentials were less than -0.20v with respect to a copper-copper sulphate reference cell. This range of values is generally considered to be indicative of non-corroding reinforcing steel.

PETROGRAPHIC EXAMINATION OF CONCRETE

Cores taken from the test slab were examined microscopically to determine the presence and distribution of wax beads in the hardened concrete. Under a polarizing microscope the spherical wax beads display an amorphous structure with internal air voids as shown in Figures 9 and 10. When incorporated into batched concrete the minute-sized wax beads look no different than clear-to amber-colored sand grains and on a cut and smoothly ground surface appear virtually indistinguishable from fine aggregate as shown in Figure 11. Therefore, for microscopic studies the wax beads must be conditioned to make them more recognizable.

When sufficient heat is applied to concrete containing wax beads to cause melting, the wax migrates into the capillary system, thus leaving a void where the wax bead existed. This is the principal mechanism involved with internally sealing of bridge decks. Generally, a visual or low-magnification examination of cores removed from heat-treated bridge slabs is sufficient to determine depth of melting. The voids resulting from the melted wax beads can be identified and measured.

In situations where the wax-bead system needs to be examined and the area is outside (below) the depth of heat penetration, the beads can be dissolved, thus making the voids easy to identify and measure. For instance, with bridge deck cores, slices may be cut from the cores and placed into a trichloroethane bath. The solvent starts dissolving

the wax within two minutes to a degree that permits identification. Figures 12 through 22 illustrate the trichloroethane etching technique and show surfaces which have been subjected to dissolution etching up to one hour.

As shown in the figures, the spherical wax beads do not necessarily remain intact when batched in concrete. Because of the shearing and agitation action of mixing the plastic concrete, the relatively soft wax beads generally break up and are found in the hardened concrete mass as variously, but typically, crescent shaped fragments (Figures 16 through 22).

In order to evaluate the distribution of the wax beads in the experimental slab at Monahans, cores were horizontally sectioned at depths of 2 1/2, 5 and 7 1/2 inches from the top surface. These sections were polished and examined microscopically by linear traverse methods as outlined in ASTM C 457. Instead of counting and measuring the entrained air void system the wax-bead voids present after dissolution etching in trichloroethane were examined. Measurements of 6.2, 5.5 and 5.2 percent "wax beads" were determined at the above-listed depths respectively. No clumping or irregular distribution of the beads within a sectioned area was observed.

Sections cut vertically in the experimental slab cores removed after the "heat blanket" treatment showed that the wax migrated into surrounding paste for the area within 1/2 inch of the slab surface. Below that depth the wax was still intact. Within the "melted" zone,

the wax voids still had a thin lining of residual material.

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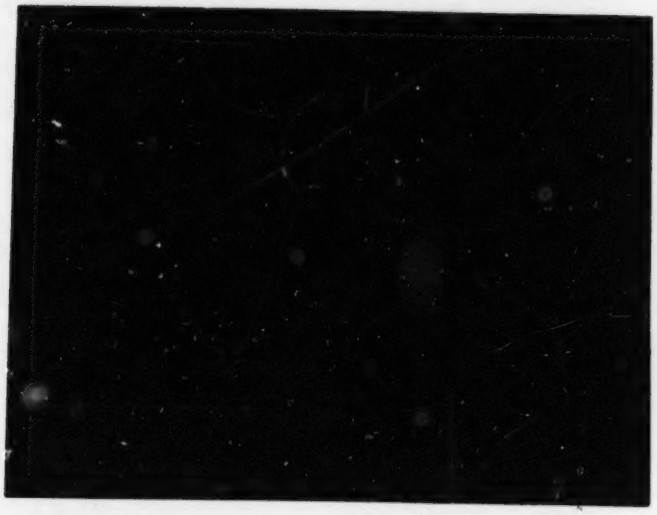

Fig. 9
"Interseal" brand wax beads.
Note internal voids.
Polarized light. Mag. 62X

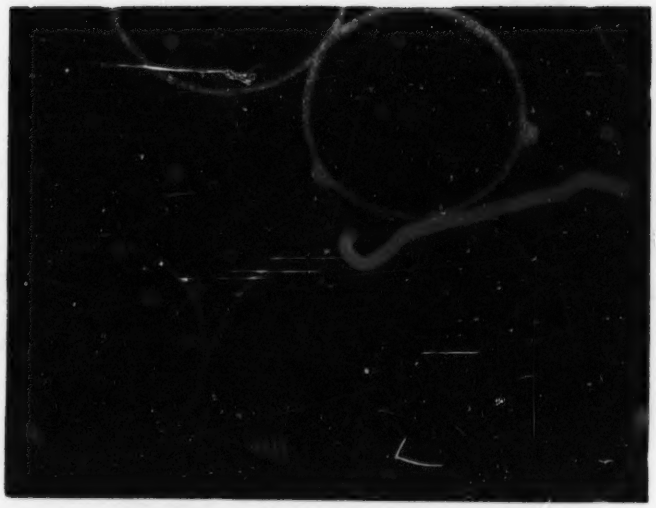

Fig. 10
Same as above.
Mag. 125X

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Wax-bead internally sealed
concrete test slab cores.
Dist. 6 Ward Co.
IH 20-1(86)082 PD9152

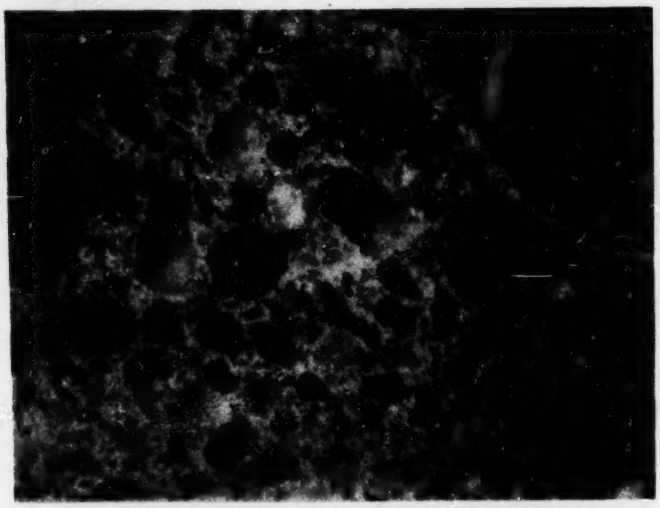

Fig. 11
Mag. 22X

Fig. 12
Two-minute dissolution
etched in trichloroethane.
Mag. 22X

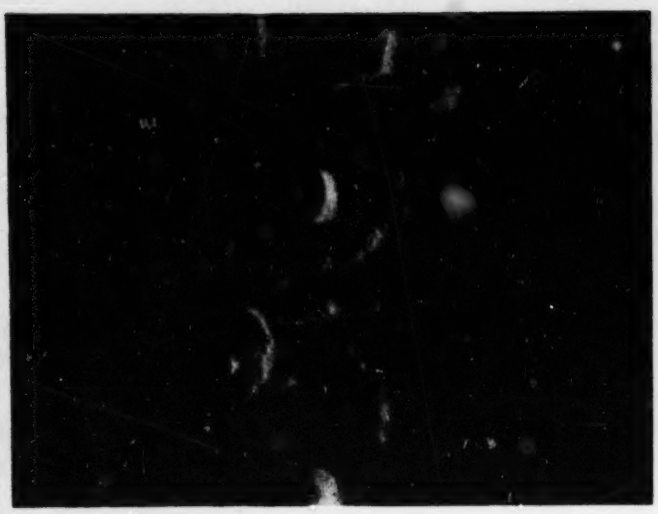

Fig. 13
Mag. 52X

Fig. 14
Mag. 105X

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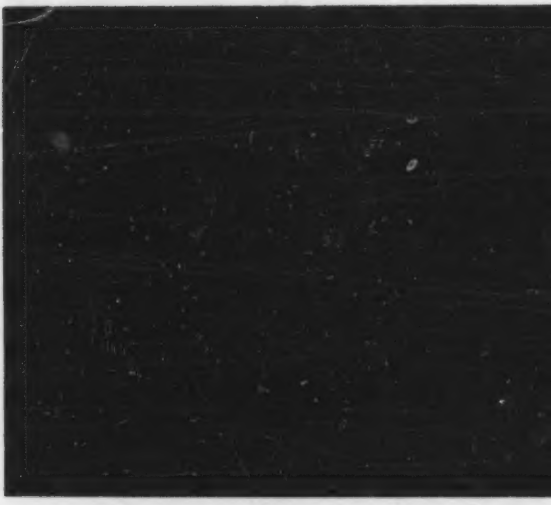

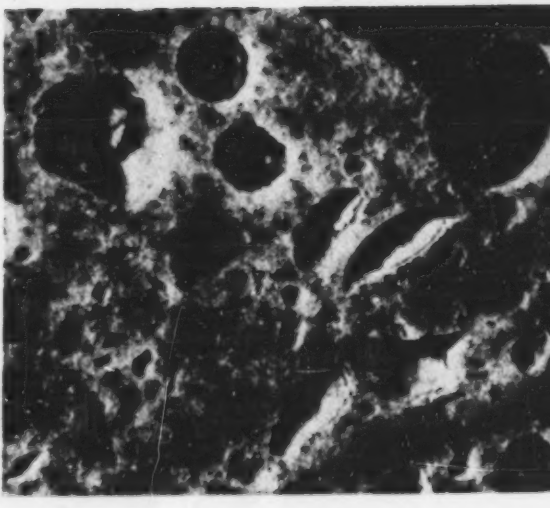

Fig. 15
Wax beads with three-minute etch in trichloroethane. Note large entrapped air void.
Mag. 8X

Fig. 16
Various shapes of wax bead fragments etched with trichloroethane.
Mag. 22X

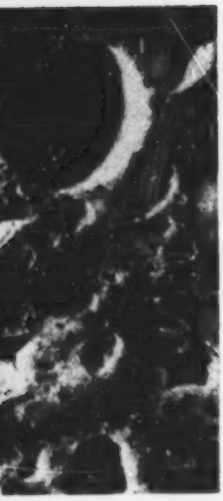

Fig. 17
Un-dissolved wax remaining in bead void space after 45-minute etch period with trichloroethane. Large sphere is air void.
Mag. 22X

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Figs. 18-19
Various shapes and sizes of wax beads partially dissolved by trichloroethane. Note peripheral shrinkage of wax bead and internal void.
Mag. 105X

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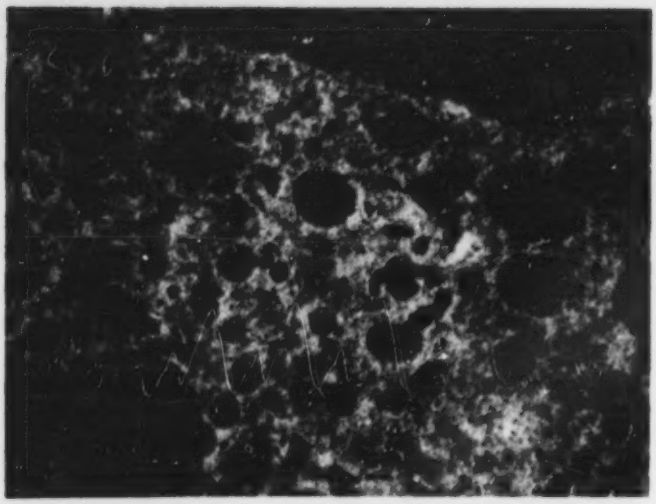

Fig. 20
Wax-bead voids after one-hour dissolution etch with trichloroethane.
Mag. 22X

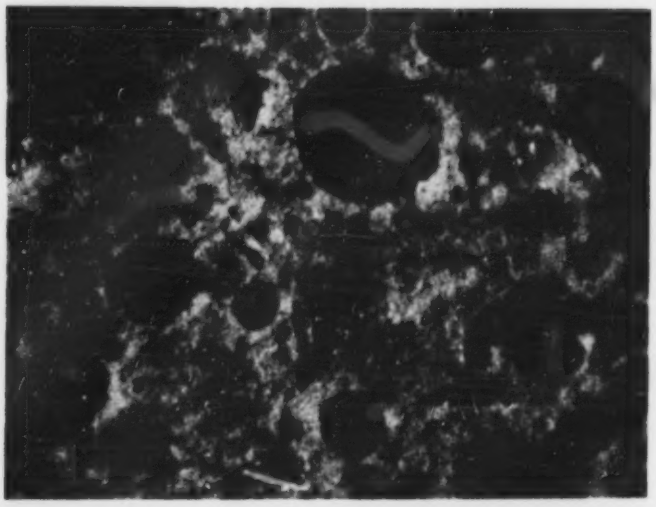

Fig. 21
Same view above. Note crescent-shape wax bead fregments.
Mag. 52X

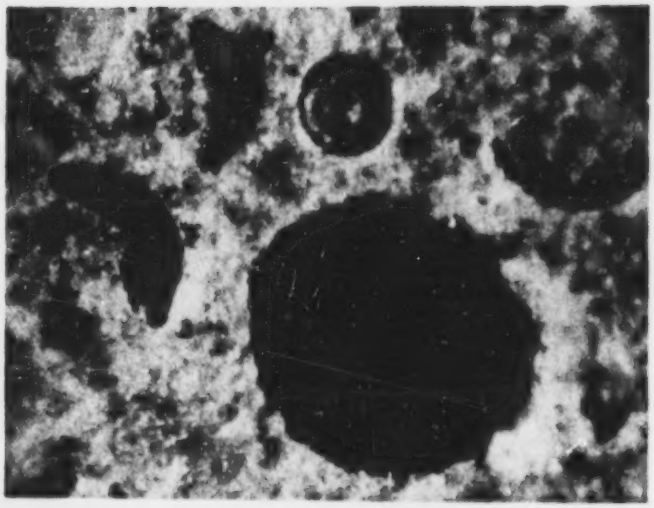

Fig. 22
Same view above. Note insoluble residue in smaller wax-bead void.
Mag. 105X

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SUMMARY AND CONCLUSIONS

The objective of this study was to construct a bridge deck impregnated with wax beads and evaluate its performance under service conditions. Heat treatment of the deck was completed in late October, 1980. The bridge, however, has not been opened to traffic as of the writing of this report.

Based on the tests conducted on a small test slab and the completed bridge deck, the following statements and conclusions are made:

1. None of the cracking observed during the crack survey can be attributed to the heat treatment.

2. The dye penetration test did not show any variation in the absorption rate of the heated zone over the unheated zone or the unheated slab concrete.

3. The freeze-thaw durability of the heat treated concrete was better than the unheated sublayers and much better than the unheated test slab concrete.

4. The chloride penetration of the heated bridge deck concrete was improved over the unheated test slab concrete, especially in the top one-half inch.

5. The corrosion potential survey showed no evidence of reinforcing steel corrosion. This is as expected since there

has been no salt applied to the deck.

6. The wax beads in the concrete caused no apparent reduction in concrete strengths. The measured strengths were equal to or greater than required for normal Class "C" bridge slab concrete.

REFERENCES

1. Implementation Package 77-9 "Internally Sealed Concrete - Guide to Construction and Heat Treatment", U.S. Department of Transportation, Offices of Research and Development, Implementation Division, April 1977.

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APPENDIX A

SPECIAL SPECIFICATIONS

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STATE DEPARTMENT OF HIGHWAYS AND PUBLIC TRANSPORTATION

SPECIAL SPECIFICATIONS

ITEM 4222

INTERNALLY SEALED CONCRETE

4222.1. Description. This item shall govern for the concrete required to construct an internally sealed concrete bridge slab.

4222.2. Materials.

(1) Concrete. The concrete shall be Class "C" complying with the Item, "Concrete for Structures", except as modified by the following:

(a) The coarse aggregate shall be either Grade 2 or Grade 3.

(b) The internally sealed concrete shall contain a minimum of 6.5 sacks of Type II cement per cubic yard; have a water-cement ratio not to exceed 5.0 gallons per sack; have a coarse aggregate factor not less than 0.70; shall be non-air entrained; and shall contain 2.1 cubic feet of wax beads per cubic yard. The wax beads shall replace an equal volume of fine aggregate or combination of fine and coarse aggregate. Approved water reducing and retarding admixtures may be used. The slump range shall be 2 to 4 inches.

(2) Wax Beads. The wax beads shall consist of a physical blend of 75 $\pm$ 5 percent parafin wax, (melt point 149 $\pm$ 2 F), and 25 $\pm$ 5 percent crude Montan Wax.

Wax beads shall be spherical in shape and have a specific gravity of 0.845 ($\pm$ 0.020), with a void volume of 8.0 to 12.0 percent. At least 60 percent of the wax beads shall have discernible voids. The gradation shall be as follows:

Sieve No.	Percent Passing
No. 20	99-100
No. 80	0-5

The Contractor shall furnish certification of compliance with the above requirements.

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The wax beads shall be packaged in moisture proof containers not to exceed 250 lbs. Maximum temperature indicators shall be placed in two of the wax bead containers for each lot or separate shipment and plainly marked. The wax beads shall be shipped and stored so that they are not exposed to temperatures in excess of 120 F. Any container of beads showing evidence of melting or agglomerating shall be rejected.

4222.3. Construction Methods. Construction of the bridge deck with internally sealed concrete shall be in accordance with all pertinent requirements of the Item "Concrete Structures" supplemented with the following requirements:

If an insulated bin is provided for the wax beads, they may be weighed and batched with the other materials. If such a bin arrangement is not provided, the wax beads shall be pre-weighed for each batch and added manually, after all other materials have been charged into the mixer, except that approximately 1/3 of the mixing water shall be withheld and then added after the beads have been introduced. The concrete shall then be mixed at mixing speed for 70 to 100 revolutions.

The internally sealed concrete shall be covered with wet burlap as soon as possible after texturing, to be followed by a ten day water cure, in accordance with the Item, "Concrete Structures". Interim membrane curing will not be permitted.

The parapet railing shall not be placed prior to completion of the heat treatment.

4222.4. Heat Treatment. Heat treatment will be accomplished with personnel and equipment of the Department after the deck has been in place at least 21 days.

Prior to heat treatment, the Contractor will be required to remove all equipment, forms, mats, and other materials from the deck. The deck shall be swept clean of any loose dirt, sand and other debris. Oil and/or grease spots shall be removed.

4222.5. Measurement and Payment. No direct measurement or payment will be made under this Item; the requirements herein being measured and paid for in accordance with the Item 4223, "Reinforced Concrete Slab".

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STATE DEPARTMENT OF HIGHWAYS AND PUBLIC TRANSPORTATION

SPECIAL SPECIFICATION

ITEM 4223

REINFORCED CONCRETE SLAB

4223.1. Description. This item shall govern the method of forming, the materials used, and the construction of a reinforced concrete slab in accordance with the details shown on the plans. The Contractor shall have the option of constructing a cast-in-place slab using conventional forms or a cast-in-place slab using permanent metal deck forms.

4223.2. Materials. All materials for concrete shall conform to the requirements of the Items, "Concrete Structures", "Concrete for Structures" and/or "Internally Sealed Concrete".

Reinforcing steel shall conform to the requirements of the Item, "Reinforcing Steel" and the details shown on the plans.

Materials for permanent metal deck forms shall be in accordance with the Item, "Permanent Metal Deck Forms".

The slab concrete for the westbound structure over Park Road 41 shall be Class "C" (Internally Sealed Concrete), for the eastbound structure shall be Class "C-C", and for the Structure at FM 1053 shall be Class "C".

4223.3. Test Slabs. After adequate trial batches have been made and tested for specification requirements on the Class "C" (Internally Sealed Concrete) and on the Class "C-C" concrete, and a minimum of 30 days prior to placing slab concrete in the structures, the Contractor shall cast a test slab from each type of concrete for evaluation. The test slabs shall be approximately 6'0" X 8'0" and 8 inches in thickness. The test slabs shall be formed on the bottom and all sides.

The test slabs shall be cast simulating actual placing conditions for the bridge slabs as nearly as possible.

The test slabs will be used to evaluate mix design; construction and vibration procedures; air-entrainment, when required; wax bead distribution, when required; and the effects of curing and texturing.

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4223.4. Construction Methods. Unless otherwise shown on the plans either of the forming systems listed herein may be used within the same span or structure.

Prior to the submission of shop drawings for beams, the Contractor shall notify the Engineer in writing as to the types and arrangements of forming and diafram options to be used.

Design and construction requirements of the standard details, and of the Items, "Concrete Structures", "Permanent Metal Deck Forms" or "Reinforcing Steel" shall govern for the particular method(s) of forming used.

When the method of forming chosen by the Contractor requires revised quantities of concrete and/or reinforcement and/or revision of elevations in order to make the system compatible with plan requirements, he shall submit proposed details to the Engineer for approval, prior to the erection of forms and placement of concrete.

4223.5. Measurement. Reinforced concrete slab(s) placed under this Item will be measured by the square foot of horizontal surface area using the nominal dimensions and configuration shown on the plans, transverse measurement being made out to out of slab, with longitudinal measurement being made between ends of units or spans. Diafram and haunch concrete and reinforcement will be considered as a portion of the slab.

Structural steel, anchor bolts, armor joints, preformed joint seal, rail (including the concrete parapet portion), concrete median barrier and median barrier rail will be measured in accordance with the Item under which they are furnished, and will not be subsidiary to this Item.

The quantities of concrete and reinforcing steel shown in the plans are estimates furnished for information only, and may or may not represent the actual amount or quantity to be placed in the slab(s), and, unless otherwise noted, are based on a conventionally formed slab. These quantities include amounts for concrete diaframs, brackets and other required attachments, and for haunch concrete when required, based on the profile grade and theoretical camber of the beams.

No additional measurement will be made for concrete or reinforcing steel due to a grade change required due to a variation in camber of the beams from theoretical camber, or for additional quantities required by optional methods of forming.

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4223.6. Payment. The quantity to be paid for shall be measured in place, as specified in Article 4223.5. above.

The unit price bid per square foot for "Reinforced Concrete Slab, (Class "C")", "Reinforced Concrete Slab (Class "C-C")", or for "Reinforced Concrete Slab, (Class "C" Internally Sealed Concrete)", shall be full compensation for furnishing, hauling, mixing, placing, curing and finishing of concrete; furnishing and placing all reinforcing steel; furnishing wax beads; furnishing water reducing plasticizer; all grouting and pointing; for casting of test slabs; furnishing and placing drains and expansion joint material except where specifically furnished under another Item; furnishing and placing metal flashing strips; for all forms (removable and/or permanent) and all falsework, and for all labor, tools, equipment, and incidentals necessary to complete the work.

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APPENDIX B

CONCRETE DESIGN WORK SHEET

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Construction Form 350

County: Ward
Project: I20-1(86)082
Date: 7-2-80

Design No: IC WAX

CONCRETE DESIGN WORK SHEET
(NATURAL AGGREGATES)

AGGREGATE CHARACTERISTICS:

	SP. GR	SSD Unit Wt. Lbs./Cu. Ft.	% SOLIDS
Fine Aggregate (FA)	2.63	108	66
Coarse Aggregate (CA)	2.49	96	62.3

Water 5 gal./sk.

Cement 6.5 sk./cu. yd.

DESIGN FACTORS:

Cement Factor (CF) 6.5 sacks per cubic yard of concrete
Coarse Aggregate Factor (CAF) .70
Water Factor (WF) 5 gal. per sack of cement
Air Factor (AF) 0 %

BATCH FACTOR:

Size of Batch (Full Size) / Yield for 1-Sk. Batch = ____ = ____

BATCH DESIGN (ONE SACK)	VOLUMES: 1-SK. BATCH (CU. FT.)		VOL. TO WT. (LB.) VOL. X 62.5 X SP. GR	1-SK. BATCH WTS.	FULL SIZE BATCH FACTOR	FULL SIZE BATCH WTS.
1. Concrete Yield = Cu. Ft. per Cu. Yd. / CF	27 / 6.5 = 4.15					
2. Volume CA = Yield X CAF X Solids	4.15 x .70 x .623 =	1.8098	x 62.5 x 2.49 =	281.65	6.5	1830.7*
3. Volume Mortar = Yield — Vol. CA	4.15 − 1.81 = 2.34					
4. Volume Water = WF / Gal. Water per Cu. Ft.	5 / 7.5 =	.666	x 62.5 x 1.00 =	41.63	6.5	270.6*
5. Volume One Sk. Cement	=	0.485	x 62.5 x 3.10 =	94.0	6.5	611
6. Volume Entrained Air = Yield X AF	____ x ____ =					
7. Volume Paste = Vol. Cem. + Water + Air	0.485 + .666 + 0 = 1.151					
8. Volume FA = Vol. Mortar — Paste — Wax Beads	2.34 − 1.151 = 1.189 − .323 =	.866	x 62.5 x 2.63 =	142.3	6.5	925.3*
	Vol. Wax Beads =	.323	x62.5x.845=	17.05	6.5	110
9. Yield (Summation of 2, 4, 5, 6 & 8 to Check No. 1 Above)	= 4.15					
10. Fine Aggregate Factor = Vol. FA / FA Solids X Vol. Mortar	.866 / 66% x 2.34 =	.866 / 1.55 = .558				

* Correct For Free Moisture or Absorption.

REMARKS: Volumes in Above Are Absolute Unless Otherwise Noted.
Water Added at Mixer Must Include the Liquid of the Admixtures.

Sp. Gr. of Wax Beads = .845

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APPENDIX C

FHWA HEAT TREATING GUIDELINES

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GUIDELINES AND FIELD OPERATIONS CHECKLIST FOR HEAT TREATING INTERNALLY SEALED CONCRETE

HEATING PARAMETERS

Limit surface temperature (at 1/16 inch depth) to maximum of 160°F in first 1.3 hours of the heat run. This procedure permits potentially damaging quantities of moisture vapor to escape before the wax starts to block capillaries and cause pressure buildup.

In addition to the above, the following limitations are applicable:

1. Limit surface temperature (at 1/16 inch depth) during initial 0.5 hour of heating, to not more than 145°F, nor less than 30°F above the ambient air temperature. These precautions are to insure removal of moisture to the extent necessary.

2. A temperature of 320°F is the maximum permitted, as a precaution against damage to the concrete due to excessive temperatures. These are monitored at equivalent 1/16 inch depth surface temperature.

Heat treating is complete when concrete has reached 185°F at a 2-inch depth in all areas being heat treated, but will not be attained in less than 5 hours.

The thermocouples used to monitor the heat applied to the concrete during heat treatments have been calibrated, and the following limitations will apply:

Time Hours	Depth	Monitor Reading °C	Corrected °C	Monitor Reading °F	Corrected °F	
0 - 0.5	Surface	80	63	176	145	Maximum
0.5 - 1.3	Surface	87	71	188.6	160	Maximum
After 1.3	Surface	166	160	330.8	320	Maximum
Heat Required	2 inches	93	85	199.4	185	Minimum

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SUPPLY ITEMS

Fiberglass Insulation Batting - 3,200 sq. ft.
(Unbacked, 4" or 6" thickness)
(Width & Length - largest available, up to 50 ft. in length)

Plastic mesh netting; e.g., such as used for draining ditch erosion control. Quantity to cover entire heating spread - 1,500 sq. ft. An additional 1,500 sq. ft. to reduce heat loss on back section is recommended.

Lumber or steel rebars - 6 to 15 foot lengths for holding insulation in place in the wind.

Plastic (polyethylene), and six 1"x10"x10' boards for walking over blanket area.

MISCELLANEOUS TOOLS

1/2-inch electrical drill and 1/2-inch masonry bits (carbide)

Lumber crayon

50-foot tape

Push broom (must be effective in sweeping debris and nails off deck)

Allen wrench set

Screw drivers (Fuller and Straight)

Pliers

Crescent wrenches

Hammer

Thermometer for accurate ambient temperature

GENERATOR SPECIFICS

Blanket power will be varied from 430 volts to 560 volts - control easily accessible.

Capacity 300 kw, 3 phase, 560 volts capability, 60 Hz.

Must be in good condition, capable of sustained heavy load in range of 300 kw.

APPENDIX D

FHWA TEST PROCEDURES

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ATTACHMENT A

DYE PENETRATION TEST TO DETERMINE SEALING EFFECTIVENESS OF THE INTERNALLY SEALED CONCRETE

1. Take three 2-inch diameter by 4-inch-deep cores from the bridge deck after heat-treatment.
2. Split cores in half.
3. Place cores in an air-circulating oven at 100°F. for 14 days.
4. Immerse cores in water containing red dye (recorder ink) and a wetting agent (calgon).
5. Soak for 14 days.
6. Then split again.
7. Observe dye penetration.

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ATTACHMENT B

Resistance of Concrete to Chloride Ion Penetration

1. Scope

1.1 This method covers the determination of the resistance of internally sealed concrete specimens to the penetration of chloride ion. It is intended for use in determining the effect of variations in the properties of the concrete on the resistance of the concrete to chloride ion penetration. This test method is not intended to provide a quantitative measure of the length of service that may be expected from a specific type of concrete.

2. Test Specimens

2.1 The specimens for use in this test shall be 4-inch or greater diameter cores taken from the internally sealed concrete bridge deck after the heat-treating process has been completed.

> Note 1 - This method contemplates the use of a minimum of four specimens for each evaluation with each core not less than 3 inches (76 mm) thick and 4 inches (102 mm) in diameter. Ponding and chloride determination procedures will be difficult to perform on smaller specimens.

2.2 Since a 21-day wet cure will have been achieved prior to the heat-treating, chloride penetration analyses shall proceed as specified in item 3.

3. Procedure

3.1 The cores shall be abraded for 0.125 inch (3.2 mm) ± 0.0625 inch (1.6 mm) from the finished surface using grinding or sandblasting techniques. No water shall be used in the abrading process and the process shall not heat any portion of the concrete to more than 100°F.

3.2 Place 0.75 inch (19 mm) high by 0.50 inch (13 mm) wide dams around the top edge of all cores except one, which will then become the control core.

3.3 All cores shall be placed in a drying room of the type specified by AASHTO: T-160, "Length Change of Cement Mortar and Concrete" at a temperature of 100°F for a period of 6 days. After drying, the cores with dams will be subjected to continuous ponding with a 0.50 inch (13 mm) deep 3 percent sodium chloride solution for 90 days. Glass plates shall be placed over the ponded solutions to retard evaporation of the solution. Additional solution shall be added if necessary to maintain the 0.50 inch depth.

3.4 After 90 days of exposure the solution shall be removed from the cores. The cores shall be allowed to dry and then the surfaces shall be wire brushed until all salt crystal buildup is completely removed.

3.5 Samples for chloride ion analysis shall then be taken from all cores in accordance with the rotary hammer procedure described in report FHWA-RD-74-5 or by dry coring (1.5 inch (38 mm) minimum diameter cores) and dry sawing. Three samples shall be obtained from each core at each of the following depths:

1/16 inch (1.6 mm) to 0.5 inch (13 mm)
0.5 inch (13 mm) to 1.0 inch (25 mm)
1.0 inch (25 mm) to 1.5 inch (38 mm)

The chloride content (parts per million by weight) of each sample shall be determined in accordance with the procedures described in attachment C.

4. Calculations

4.1 The baseline chloride ion content for the test specimens shall be determined as the average chloride ion content of samples obtained from the 1/16 inch (1.6 mm) to 0.5 inch (13 mm), 0.5 inch (13 mm) to 1.0 inch (25 mm), and 1.0 inch (25 mm) to 1.5 inch (38 mm) depths within the core that was not ponded with 3 percent NaCl solution.

4.2 The absorbed chloride ion content of each sample from the ponded cores shall be determined as the difference between the total chloride ion content of that sample and the baseline value calculated in Section 4.1. If the result is less than zero, the result shall be reported as zero. The average chloride ion absorbed at each sampling depth shall be calculated.

5. Report

5.1 Reporting shall include: (1) each total chloride ion value determined in Section 3.5; (2) the average and maximum baseline chloride ion (Section 4.1); (3) each calculated absorbed chloride ion value determined in Section 4.2; and (4) the average and maximum absorbed chloride ion values calculated in Section 4.2 for each depth.

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ATTACHMENT C

STANDARD METHOD OF SAMPLING AND TESTING FOR TOTAL CHLORIDE ION IN CONCRETE

1. Scope

1.1 This method covers a procedure for the determination of the total chloride ion content of aggregates, portland cement, mortar, or concrete. The method is limited to material that does not contain sulfides, but the extraction procedure, paragraphs 5.1 through 5.6, may be used for all such materials.

2. Apparatus

2.1 Samples may be obtained by one of two methods, 2.1.1 or 2.1.2

2.1.1 Core drill.

2.1.2 Rotary impact type drill with a depth indicator and drill or pulverizing bits of sufficient diameter to provide a representative sample of sufficient size for testing.

2.1.2.1 Sample container capable of maintaining the sample in an uncontaminated state.

2.1.2.2 Spoons of adequate size to collect the sample from the drilled holes.

2.1.2.3 A "blow out" bulb or other suitable means or removing excess pulverized material from the hole prior to redrilling operations.

2.1.2.4 A pachometer capable of determining the location and depth of steel reinforcement to $\pm$ 1/8 inch ($\pm$3 mm).

2.2 Testing

2.2.1 Chloride ion or silver/sulfide ion selective electrode and manufacturer-recommended filling solutions.

Note: Suggested electrodes are the Orion 96-17 Combination Chloride Electrode or the Orion 94-6 Silver/Sulfide Electrode or

equivalents. The Silver/Sulfide electrode requires use of an appropriate reference electrode (Orion 90-92 or equivalent).

2.2.2 A millivoltmeter compatible with the ion electrode.

Note: Suggested millivoltmeter is the Orion Model 701A Digital pH/m_V meter or equivalent.

2.2.3 Magnetic stirrer and teflon stirring bars.

2.2.4 Burette with 0.1 mL graduations.

2.2.5 Balance sensitive to 0.0001 gram with minimum capacity of 100 grams.

2.2.6 Balance sensitive to 0.1 gram with minimum capacity of 1 Kg.

2.2.7 Hot plate, 250° to 400°C heating surface temperature.

2.2.8 Glassware - 100 to 250 mL beakers, filter funnels, stirring rods, watch glasses, dropper; mortar and pestle; wash bottles.

2.2.9 Sieve, U.S. Standard 50 mesh.

2.2.10 Whatman No. 40 and No. 41 filter papers (or equivalent)

Note: If equivalent filter papers are used, they should be checked to confirm that they do not contain chloride which will contaminate the sample.

3. Reagents

3.1 Concentrated HNO_3 (sp. gr. 1.42)

3.2 Sodium chloride, NaCl, reagent grade (primary standard).

3.3 Standard 0.0100 N NaCl solution. Dry reagent grade NaCl in an oven at 105°C. Cool, in a desicator, weigh out 0.5844 gram, dissolve in distilled H_2O, and transfer to a 1-liter volumetric flask. Make up to the mark with distilled H_2O and mix.

3.4 Standard 0.01 N $AgNO_3$. Weigh 1.7 grams of reagent grade $AgNO_3$, dissolve in distilled H_2O, filter into a 1-liter brown grass bottle, fill, and mix thoroughly. Standardize against 25.00 mL of the NaCl solution by the titration method given in paragraph 5.7.

3.5 Distilled water.

3.6 Methyl orange indicator.

3.7 Ethyl alcohol, technical grade.

4. Method of Sampling

4.1 Determine the depth within the concrete for which the chloride content is desired. Use the pachometer to determine reinforcement bar location and depth.

4.2 Core Method - Drill the core to chosen depth and retrieve.

4.2.1 When samples are received in the laboratory in other than pulverized condition, the sample shall be crushed and ground to a powder. All sawing or crushing shall be done dry (i.e. without water). All material shall pass a No. 50 mesh sieve. All pulverizing tools and sieves shall be washed with ethyl alcohol or distilled water and shall be dry before use with each separate sample (see note para. 4.3.7).

4.3 Pulverizing Method

4.3.1 Set the rotary hammer depth indicator so that it will drill to 1/4 inch (6 mm) above the desired depth.

4.3.2 Using a drill or pulverizing bit, drill until the depth indicator seats itself on the concrete surface.

4.3.3 Thoroughly clean the drilled hole and surrounding area utilizing the "blow out" bulb or other suitable means.

4.3.4 Reset the depth indicator to permit 1/2 inch (13 mm) additional drilling.

4.3.5 Pulverize the concrete until the depth indicator again seats itself on the concrete.

Note: Care must be exercised during this pulverizing operation to prevent the drill bit from abrading concrete from the sides of the hole above the sampling depth. To insure against this, some users utilize an 0.25 inch (6 mm) smaller diameter bit in this step than that used in para. 4.3.2.

4.3.6 Collect at least 10 grams of the material remaining in the hole using a spoon and place in the sample container.

4.3.7 If the sample, as collected, does not completely pass a 50 mesh screen, additional pulverizing shall be performed until the entire sample is finer than 50 mesh.

Note: During sample collection and pulverizing, personnel shall use caution to prevent contact of the sample with hands, or other sources of body perspiration or contamination. Further, all sampling tools (drill bits, spoons, bottles, sieves, etc.) shall be washed with ethyl alcohol or distilled water and shall be dry prior to use on each separate sample. Ethyl alcohol is normally perferred for washing because of the rapid drying which naturally occurs.

5. Procedure

5.1 Weigh to the nearest milligram a 3 gram powdered sample representative of the material under test.

Note: Some users dry the sample to constant weight in a 105°C oven and determine the dry sample weight prior to analysis. This optional procedure provides a constant base for comparison of all results by eliminating moisture content as a variable. It is generally believed that drying is only necessary when very high accuracy is desired (see Reference 1 for data in this area).

Transfer the sample quantitatively to a mortar; add 10 mL of hot (90° to 100°C) distilled H_2O to the mortar, swirling to bring the powder into suspension.

Carefully grind the slurry with a pestle until all lumps are gone. Very little grinding will be necessary for soft aggregates, but considerable effort will be required for samples containing hard aggregates.

Note: Sample particle size after grinding should be such that it will pass a 100 mesh screen. Further, about 75 percent of a properly ground sample will pass a 200 mesh screen. It is suggested that the analyst grind several trial samples, in accordance with the above procedure and then dry the samples and determine the particle size as a means of defining the grinding required for actual samples.

5.2 Transfer the slurry quantitatively from the mortar through a funnel into a 100 mL beaker, rinsing the funnel lightly with hot distilled H_2O. Add 3 mL concentrated HNO_3 to the mortar and stir with the pestle to completely dissolve any cement left in the mortar. Transfer the contents of the mortar through the funnel while continuously stirring the beaker with a glass stirring rod. Rinse the mortar, pestle, inside of the funnel and the tip of the funnel with hot distilled H_2O.

Note: Too rapid transfer of the acid into the 100 mL beaker will cause excessive foaming or frothing of samples with calcareous aggregates or organic components and resultant risk of sample loss.

5.3 Make up the solution in the 100 mL beaker to approximately 50 mL with hot distilled H_2O. Stir thoroughly to ensure complete sample digestion. Add five drops of methyl orange indicator and stir. If yellow to yellow-orange color appears solution is not sufficiently acidic. Add additional concentrated HNO_3 dropwise with continuous stirring until a faint pink or red color persists in the solution. Cover with a watch glass, retaining the stirring rod in the beaker.

Note: Due to the presence of relatively insoluble materials in the sample, the solution generally will have a strong gray color, making the detection of the indicator color difficult at times. Running of several trial samples is suggested to give the analyst practice in detecting the indicator color.

5.4 Bring the solution in the covered 100 mL beaker to a boil on a medium heat (250 to 400°C) hot plate, and then boil for a full minute with care to avoid frothing and spillovers. Remove from heat.

Note: The analysis can be stopped at this point and the sample allowed to cool in an HCl fume-free area if it is necessary. Before proceeding to the next step, however, the solution must again be brought to a boil.

5.5 Prepare a funnel fitted with double filter paper (Whatman No. 41 over No. 40 filter paper or equivalents) and a 250 mL beaker to receive the filtrate. Carefully lift the watch glass from the 100 mL beaker without tilting it, and wash any adhering drops into the filter paper with hot distilled water. Then filter the hot solution into the 250 mL beaker. Proceed carefully, employing the stirring rod to aid quantitative transfer of the solution into the filter funnel. Wash the inside of the 100 mL beaker and the stirring rod twice with hot distilled H_2O. Transfer the washings through the filter into the 250 mL beaker. Finally, carefully wash the outside of the pouring lip of the 100 mL beaker with hot distilled H_2O into the filter.

5.6 Wash the filter paper five to ten times with hot distilled H_2O, being careful not to lift the paper away from the funnel surface. Finally, lift the filter paper carefully from the funnel and wash the outside surface of the paper with hot distilled H_2O; then wash the tip of the funnel. The final volume of the filtered solution should be 125 to 150 mL. Cover with a watch glass and allow to cool to room temperature in an HCl fume-free atmosphere.

5.7 Two alternate methods are available to determine the Cl- content of the solution. Both methods utilize an ion selective electode (Cl- or Ag+) and both methods for the purpose of this analysis give results of essentially equal accuracy and precision. However, Method II offers a substantial decrease in time required for analysis over Method I.

5.7.1 Alternate Method I: Potentiometric Titration

Fill the Cl- or the Ag+ electode with the solution(s) recommended by the manufacturer, plug it into the millovoltmeter (preferably the type with a digital rather than a dial readout), and determine the approximate equivalence point by immersing the electrode in a beaker of distilled H_2O. Note the approximate millivoltmeter reading (which may be unsteady in H_2O.

Take the cooled sample beaker from 5.6 and carefully add 4.00 mL of 0.01 N NaCl, swirling gently. Remove the beaker of distilled H_2O from the electrode, wipe the electrode with absorbent paper, and immerse the electrode in the sample solution. Place the entire beaker-electrode assembly on a magnetic stirrer and begin gentle stirring.

Using a calibrated buret, add gradually and record the amount of standard 0.01 N $AgNO_3$ solution necessary to bring the millivoltmeter reading to -40 mv of the equivalence point determined in distilled H_2O. Then add standard 0.01 N $AgNO_3$ solution in 0.10 mL increments recording the millivoltmeter reading after each addition.

As the equivalence point is approached, the equal additions of $AgNO_3$ solution will cause larger and larger changes in the millivoltmeter reading. Past the equivalence point, the changes per unit volume will again decrease. Continue the titration until the millivoltmeter reading is at least 40 mv past the approximate equivalence point.

The endpoint of the titration usually is near the approximate equivalence point in distilled water and may be determined by (1) plotting the volume of $AgNO_3$ solution added versus the millivoltmeter readings. The endpoint will correspond to the point of inflection of the resultant smooth curve, or (2) calculating the differences in millivoltmeter readings between successive $AgNo_3$ additions and calculating the total volume of $AgNO_3$ which corresponds with each difference (i.e., the midpoints between successive additions).

Example:

Raw Data		Difference	
Titrant Volume	Millivolt Reading	Titrant Midpoints	Millivolt Differences
4.2 mL	130.0	4.25 mL	5.0
4.3 mL	135.0	4.35 mL	7.0
4.4 mL	142.0	4.45 mL	10.0
4.5 mL	152.0	etc.	
etc.			

The endpoint will be near the midpoint which produced the largest change in millivoltmeter reading. It may be determined by plotting midpoints versus differences and defining the $AgNO_3$ volume which corresponds to the maximum difference on a smooth, symmetrical curve drawn through the points. However, it can usually be estimated accurately without plotting the curve by choosing the midpoint which corresponds to the maximum difference and adjusting for asymmetry, if any. In other words, if the differences on each side of the largest difference are not symmetrical, adjust the endpoint mathematically in the direction of the larger differences. Detailed examples of this adjustment are contained in Reference 1.

5.7.2 Alternate Method II: Gran Plot Method

This method is comparable with either a Cl- or Ag+ ion-selective electrode. Attach the electrode of choice to a compatible digital millivoltmeter after filling with required solutions as per the electrode manufacturer's instructions. Clean the electrode with distilled H_2O and pat dry with absorbent paper.

Weigh the cooled sample and beaker from 5.6 without the watch glass and record the weight. Using a calibrated buret, titrate the sample to 225 mv $\pm$ 5 mv (Cl- electrode) or 310 $\pm$ 5 mv (Ag+ electrode) with standard 0.01N $AgNO_3$ solution. Record the volume added and the millivoltmeter reading.

Continue to titrate in 0.50 mL increments recording the volume added and the millivoltmeter reading for each increment. Add and record the data for at least five increments. Empty, clean, dry, and weigh the beaker. Subtract beaker weight from beaker + solution weight determined above to define solution weight.

Additional information on the Gran Plot Method is given in Reference 2.

6. Calculations

6.1 Alternate Method I - Potentiometric Titration

Determine the endpoint of the titration as described in para. 5.7.1 by either plotting a curve or estimating from the numerical data. Calculate the percent Cl- ion from the equation:

$$\% \text{ Cl} = 3.5453 \left(\frac{V_1 N_1 - V_2 N_2}{W} \right)$$

Where:

V_1 = endpoint in mL

V_2 = Volume of NaCl solution added, in mL

N_1 = normality of AgNO

N_2 = Normality of NaCl solution

W = Weight of original concrete sample in grams.

6.2 Alternate Method II - Gran Plot Method

Calculate corrected values for each of the volumes recorded in 5.7.2 by the equation:

$$V_{correct} = \frac{V \text{ record}}{W/100}$$

Where:

W = original solution weight in grams

V record = Volumes recored in mL

If any of the V correct values are greater than 10, see para. 6.3. If less than 10, plot these corrected values versus the corresponding millivolt readings on Orion Gran Plot Paper (10 percent volume corrected type with each major vertical scale division equal to 5 millivolts) or equivalent. Draw the best straight line through the points and read the endpoint at the intersection of the line with the horizontal axis of the graph. Calculate the actual endpoint by the equation:

$$E_a, \text{ ACTUAL ENDPOINT} = E_g \left(\frac{W}{100} \right)$$

Where:

E_g = Endpoint determined from graph in mL

W = Weight of solution in grams

Then $$\% \text{ Cl-} = \frac{3.5453}{Wc} E_a N$$

Where:

E_a = Actual endpoint, in mL

N^a= Normality of $AgNO_3$

W_c = Concrete sample weight in grams

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6.3 Supplementary Gran Method Calculations:

When the V correct volumes determined in 6.2 are greater than 10, discard the values and follow the following procedure.

Choose a constant which, when subtracted from all V record volumes, yields values less than 10 ml.

Note: This constant, designated as X in the formulas below, is normally assigned an even value such as 5, 10, 15, 20, etc.

Calculate a revised solution weight W_r

$$W_r = W + X$$

Where: W = original solution weight in grams
X = the constant

Then calculate corrected volumes for each recorded volume as:

$$V \text{ corrected} = \frac{V \text{ record} - X}{W_r/100}$$

Plot these values and determine the graph endpoint E_g as described in para. 6.2. The actual endpoint, E_a is then:

$$E_a = E_g \left(\frac{W_r}{}\right) + X$$

Where: E_a = actual endpoint in ml
E_g = endpoint from graph in ml
W_r = revised solution weight in grams
X = the constant chosen above

Calculate the chloride content using the formula given in para. 6.2.

6.4 The percent chloride may be converted to pounds of Cl- per cubic yard of concrete as follows:

$$\text{lbs. Cl-/yd}^3 = \%Cl = \left(\frac{UW}{100}\right)$$

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Where:

UW = Unit weight of concrete per cubic yard.

Note: A unit weight of 3915 lbs/yd^3 is often assumed for normal structural weight concrete when the actual unit weight is unknown.

7. References

1. Clear, K. C., "Evaluation of Portland Cement Concrete for Permanent Bridge Deck Repair," Federal Highway Administration, Report FHWA-RD-74-5, February 1974. National Technical Information Service PB 232-604/AS.

2. Clemena, G. C., Reynolds, J. W., and McCormick, R. M., "Comparative Studies of Chloride in Hardened Concrete," Virginia Highway and Transportation Research Council, Report 77-R7, August 1976. This report is available from the National Technical Information Service, Springfield, Virginia 22161.

ATTACHMENT D

Procedure for Determining Percent Wax Beads by Volume in Plastic Concrete

1. Equipment Required:

   a. Chace Air Meter (Calibrated as per attachment).
   b. Squeeze bottle of saturated NaCl solution
   c. Squeeze bottle of H_2O
   d. Screen: #4 mesh

2. Since wax beads have a lower specific gravity than the saturated NaCl solution, they will float. Due to this fact, the Chace Air Meter can be utilized to determine the percent wax beads by volume in plastic concrete.

3. Procedure:

   a. Screen a sample (approximately 1 lb.) of the plastic concrete through a #4 mesh screen to remove the coarse aggregate.

   b. Fill the sample cup of the Chace Air Meter with a sample of the screened plastic concrete.

   c. Remove the excess plastic concrete from the sides of the sample cup and stopper with a squeeze bottle of H_2O (i.e., rinse the <u>outside</u> of the sample cup and stopper with water).

   d. Place the sample cup containing the concrete sample inside the Chace Air Meter. Make sure that the stopper is in tightly.

   e. Fill the meter through the narrow neck with saturated NaCl solution until the solution level reaches the bottom of the <u>narrow</u> neck on the meter.

Fill with NaCl solution to here.

   f. Holding the opening at the top of the meter closed (with thumb or forefinger), vigorously shake the meter for 30 seconds separating the wax beads from the plastic concrete.

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g. Add NaCl solution through the neck of the meter until the top of the wax bead column is even with the top mark on the neck of the meter. (Meter must be vertical.)

h. Holding the meter vertically, gently tap the rubber stopper against a solid surface for a period of 15 seconds. This will cause any stray beads to float up into the neck of the meter.

i. Allow the meter to set on flat surface for a period of 1 minute (note: meter must be setting on its rubber stopper in the vertical position).

j. Determine the height of the bead column to the nearest 1/10 of a division by counting the number of divisions that the column occupies in the neck of the meter.

k. To determine the percent wax beads by volume in the mix, simply convert the meter column height reading to percent wax beads by using your calibration curve.

l. Note: In order to obtain an accurate percent wax bead value, it is necessary to run three samples using the average of the three as the actual percent wax beads in the concrete mixture.

Calibration of Chace Air Meter to Read Percent Wax Beads by Volume

1. Since all Chace Meters do not yield the results for the same sample mix, it is necessary to calibrate the meter that will be used for wax bead volume determinations. Also, since the calibration procedure is time-consuming (approximately 3 hours), it is suggested that 1 meter be set aside and used exclusively for wax bead volume determinations (Note: This eliminates the need for continual recalibration because the same meter will be used each time.)

2. To calibrate the meter, four mix designs are needed: 5 percent wax beads by volume, 6 percent wax beads by volume, 8 percent wax beads by volume, and 10 percent wax beads by volume. Also, a total mix weight of approximately 2600 grams is desirable to facilitate hand mixing. Use the following table to design those mixes. (Note: The table is based on: CF = 7 bags/yd^3, w/c = 0.55, air = 3 percent.)

Solid Volumes in $ft.^3/yd^3$

Percent Wax	5	6	8	10
Cement	3.35	3.35	3.35	3.35
Air	0.81	0.81	0.81	0.81
Water	5.80	5.80	5.80	5.80
Fine Aggregate	8.22	7.95	7.41	6.87
Coarse Aggregate	7.47	7.47	7.47	7.47
Wax Beads	1.35	1.62	2.16	2.70

To obtain the S.S.D. weight of each material for a small (2600± grams) batch that can be hand mixed easily, multiply each volume in the table by the following:

S.S.D. weight of material in grams = (43.00)x(S.G.)x(Volume from table)

Constant ↑

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Where:

S.G. = Specific gravity of material
Volume from table = Volume of that material for the percent wax beads desired. (Example: For a 4 percent wax bead mix, the cement volume is 3.35 ft.^{3}).

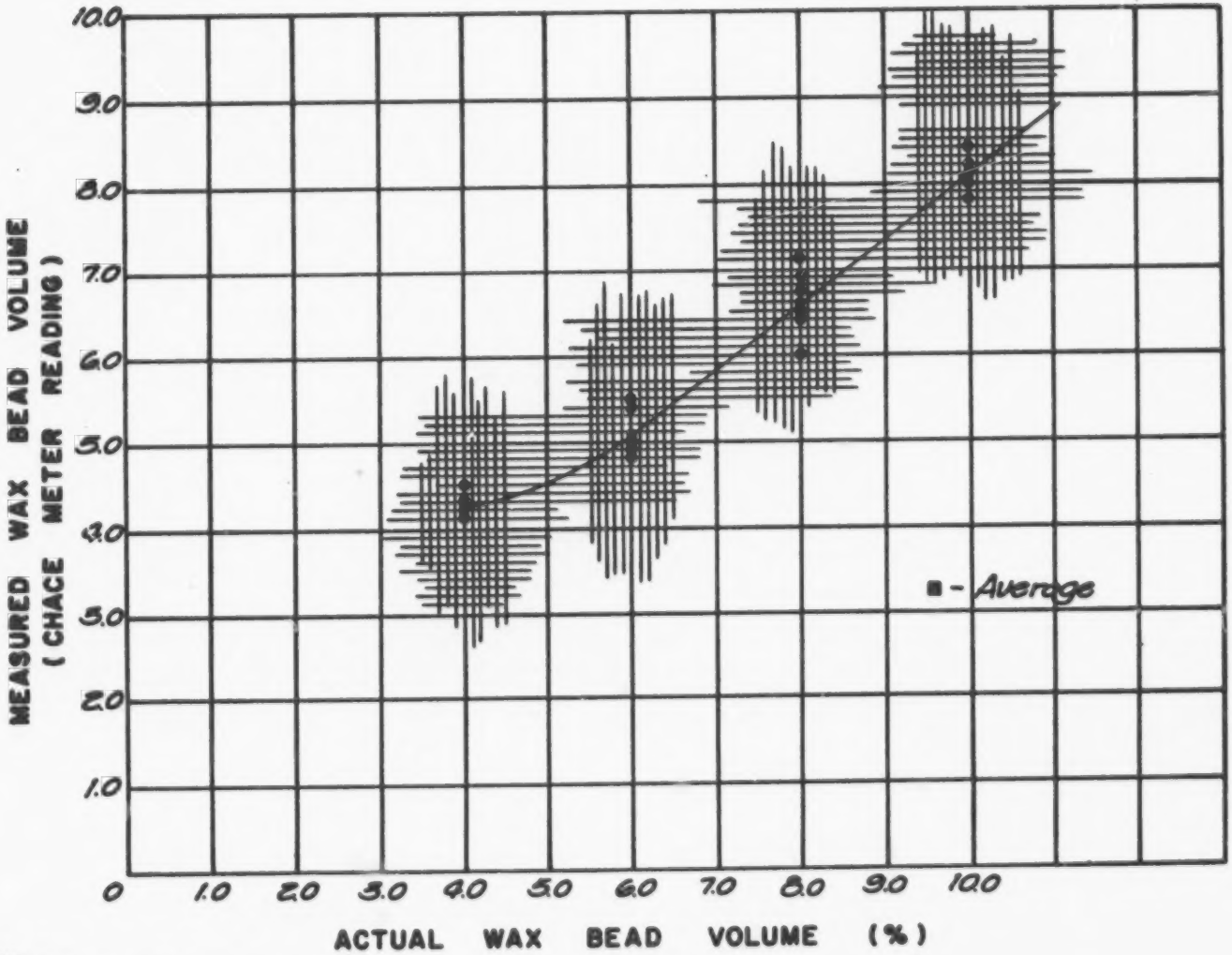
Next, obtain the batch weights and adjust for moisture in the aggregates as necessary to obtain <u>final</u> mix weights.

*Note: A sample for a 5 percent by volume wax bead mix is attached. Do not use this as a standard mix, it is only for reference.

3. Calibration Procedure:

   a. Prepare the first mix (5 percent wax beads by volume).
   b. Follow the instructions, (Attachment D.3. - "Procedure for Determining Percent Wax Beads by Volume in Plastic Concrete") through step J. (Note: Do the procedure four times to obtain four readings.)
   c. Follow steps a and b for the other three mixes (i.e., 6 percent, 8 percent, 10 percent).
   d. Average the four readings for each mix.
   e. Plot the average readings against the actual percent wax beads in each mix to obtain the calibration curve.
   f. Make up a conversion sheet that can be kept with the meter for field use.
   g. Samples of the calibration curve and conversion sheet follow.

4. Once the meter has been calibrated, it may be used in the field to monitor wax bead content (percent by volume) while the concrete is being placed.



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WAX BEAD VOLUME CONVERSION

Chace Meter Column Height	Actual Percent Wax Bead Volume
4.10	4.75
4.29	5.00
4.49	5.25
4.68	5.50
4.87	5.75
5.06	6.00
5.25	6.25
5.43	6.50
5.64	6.75
5.82	7.00
6.02	7.25
6.20	7.50
6.40	7.75
6.59	8.00
6.77	8.25
6.96	8.50
7.15	8.75
7.34	9.00
7.54	9.25
7.72	9.50
7.91	9.75
8.10	10.00

Sample 5 Percent by Volume Wax Bead Mix

C.F. = 7 bags/yd^3 | W/C = 0.55 percent | 3 percent Air | No Admixture
3 percent total moisture in sand | 0 percent absorption in sand | 3 percent free H_2O in sand

0.5 total moisture in stone | 0.5 percent absorption in stone | 0.0 percent free H_2O in stone

Specific Gravities: Cement = 3.15, Sand (F.A.) = 2.65, Stone (C.A.) = 2.77, Wax Beads = 0.84

SSD Weights (Grams)

Cement	= 453.76	
Air	= -	
Water	= 249.4	
Fine Aggregates	= 936.67	
Coarse Aggregates	= 889.75	
Was Beads	= 48.76	
TOTAL WT.	2578.34	

Final Batch Weights (Grams)

Cement	= 453.76		
Air	= -		
Water	= 225.98	-----	[249.4 - (960.09 - 936.67)]
Fine Aggregate	= 960.09	-----	[(936.67)(1.025)]
Coarse Aggregate	= 889.75		
Wax Beads	= 48.76		
TOTAL WT.	2578.34		

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